



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



(11) **EP 1 033 141 A1**

(12)

## EUROPEAN PATENT APPLICATION

(43) Date of publication:  
06.09.2000 Bulletin 2000/36

(51) Int Cl.7: **A61L 15/00, A61L 15/28,  
A61L 15/58, C08J 9/30**

(21) Application number: 00301693.8

(22) Date of filing: 02.03.2000

(84) Designated Contracting States:  
**AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU  
MC NL PT SE**  
Designated Extension States:  
**AL LT LV MK RO SI**

(72) Inventors:  
• **Fattman, George**  
New Jersey 08054 (US)  
• **Bayless, Richard**  
New Jersey 08853 (US)

(30) Priority: 02.03.1999 US 122364 P

(74) Representative: **Holmes, Miles Keeton et al**  
**D. YOUNG & CO.,**  
21 New Fetter Lane  
London EC4A 1DA (GB)

(71) Applicant: **BRISTOL-MYERS SQUIBB COMPANY**  
Princeton, NJ 08543-4000 (US)

(54) **Hydrocolloid adhesive**

(57) The invention is a foamed, pressure sensitive, hydrocolloid adhesive for bonding to the body, and methods for its manufacture. The foamed structure of the adhesive itself improves absorbency, enables trans-

mission of moisture through the adhesive, increases flexibility, and lowers product cost, all of which are key elements in making a product that is effective in adhering to the body.

EP 1 033 141 A1

## Description

BACKGROUND OF THE INVENTION

5 [0001] Adhesion to the body is a complicated process that is necessary to the functionality of a wide variety of products including but not limited to wound care dressings and ostomy appliances. It is known that adhesion to the skin for prolonged periods of time requires management of perspiration and other trans-epidermal water losses (TEWL). Pressure sensitive adhesives containing water-absorbing powders have been found effective in TEWL management. In particular, hydrocolloid adhesives are especially effective, and are preferred for this purpose.

10 [0002] It is common practice to manage wounds by covering them with a hydrocolloid wound dressing. These dressings typically consist of a pressure sensitive adhesive into which has been dispersed hydrocolloid particles. US Patents 3,339,546, 4,551,490, and others comprising the prior art of hydrocolloid containing pressure sensitive adhesives demonstrate this practice. Hydrocolloid wound dressings are exceptional in their ability to manage wound exudate. They achieve long wearing times, and provide a moist wound healing environment. This environment promotes autolytic debridement of the wound, non-traumatic dressing removal, and many other improvements over conventional adhesives and dressings that are believed to be beneficial to wound healing.

15 [0003] While hydrocolloid adhesives are known for the valuable characteristics described above, one limitation is their short-term rate of absorption. Typically the moisture absorbing capacity of a hydrocolloid adhesive is exceptional, and can exceed 5000 grams per square meter per day (gsm/d). However, absorption during the first hour is typically below 1000 gsm/d, and proportionally less at shorter times. As a result, hydrocolloid wound dressings are effective for a certain range of wounds, which produce light to moderate exudate. For heavily exuding wounds, the inability of hydrocolloid dressings to manage exudate leads to leakage of wound fluid sometime during the first 24 hours of wear.

20 [0004] For heavily exuding wounds, foamed polymers coated with a pressure sensitive adhesive on their wound contact surface are sometimes used. It is believed that their familiar design (similar to strip bandages or gauze) and ability to absorb wound exudate relatively quickly are factors in their selection. However, these products suffer from several limitations. Unlike hydrocolloid wound dressings, these products are not noted for the preferred wound healing capabilities of hydrocolloid dressings. Further, they typically have limited long term absorbing capacity as their absorbency results primarily from a mechanical effect produced by the pores and cells of their foamed structure. Obviously, an additional limitation of conventional foamed dressings is that they will lose moisture when compressed. Finally, as the wound contact surface may be coated with a pressure sensitive adhesive to adhere to the body, the wound may be traumatized upon removal of the conventional foam dressing when that adhesive bonds to the healing wound itself.

SUMMARY OF THE INVENTION

35 [0005] Aspects of the invention are defined in the claims.

[0006] Broadly speaking, one aspect of the invention comprises introducing gas bubbles into a hydrocolloid adhesive.

[0007] In another aspect, the invention provides a foamed hydrocolloid adhesive.

40 [0008] The invention described herein improves the state of the art for hydrocolloid adhesives by changing the structure of the adhesive itself. Gas bubbles introduced into the pressure sensitive adhesive during the manufacturing process create a lightweight, cellular, two-phase structure. Properties of the foamed material depend on the individual properties of both the gas and solid phases. The size and extent of each phase are key characteristics in describing the structure of the foamed adhesive. In particular, the size and shape of cells in the gas phase determine the ultimate properties of a foamed structure for a given solid phase. Additionally, the density of the overall foamed structure can be useful for characterization. The structure of the foam may be open celled, with two or more cells connected by

45 tunnels, or closed celled, where cells are predominantly isolated from one another.

[0009] Foaming hydrocolloid adhesives increases their flexibility and lowers the density and cost of the overall adhesive product. Creation of the foamed structure also results in an increase in surface area of the adhesive exposed to the contact surface. The increased surface area is particularly useful for adhesion to the body as it enables rapid absorption of liquids by a mechanical means. The time scale for absorbing liquids by physically trapping them in the cells of the foamed adhesive is significantly shorter than that for absorbing liquids chemically by the forces of attraction in the hydrocolloids themselves. Further, when liquids are trapped in the cells of the foam, the area of contact between the absorbed liquid and the solid phase of the adhesive is increased. As a result, a second, chemical based absorption process begins. These two processes work together to increase the overall absorption rate for a foamed hydrocolloid adhesive.

50 [0010] Depending on the size, shape, and extent of the cells, the foaming process can create a structure where a tortuous path of open cells and tunnels connects two exterior surfaces of a foam. In that case, moisture from one surface can rapidly be conducted to another surface. In the case of a wound dressing, this process can significantly enhance the overall rate and capacity for management of TEWL. Wound fluid quickly fills the cells of the foamed

adhesive and is then conducted to another surface of the adhesive. If that surface is exposed to the atmosphere, the fluid can evaporate. If that surface is laminated with a film capable of transmitting moisture or moisture vapor, then wound exudate can pass through that film. In either case, fluid is managed at a faster rate and to a greater extent (capacity) than is possible using unfoamed hydrocolloid dressings. Extension of the concept of a foamed hydrocolloid adhesive to other kinds of body adhesion is clear to those skilled in the art. For example, improvements resulting from the foamed structure enable the adhesive to manage a greater quantity of perspiration generated at a faster rate than an unfoamed product. This improvement is useful in general for adhesion to the body, and in particular for wound dressings and ostomy appliances as cited above.

[0011] As an ostomy device, a foamed hydrocolloid adhesive skin barrier offers several benefits over the state of the art. Flexibility of the skin barrier is greatly enhanced compared with a non-foamed barrier of the same composition. A secure attachment to the skin can be achieved, while reducing the removal force by virtue of the lessened contact area. As for wound dressings, intimate contact between hydrocolloid adhesives and the skin of an ostomate is well established as a beneficial attribute. Finally, the ability of the foamed structure to conform to irregular surfaces of the body to which an ostomy device must be attached is improvement over non-foamed adhesives.

#### DETAILED EMBODIMENT OF THE INVENTION

[0012] Foamed hydrocolloid adhesives can be achieved by introducing gas bubbles into the pressure sensitive hydrocolloid adhesive. There are two preferred methods for accomplishing this introduction. First, chemical blowing agents or other materials added to the adhesive formula itself may generate gas bubbles by a variety of mechanisms. These mechanisms include but are not limited to chemical reaction, physical changes (for example, the breaking of microspherical particles containing the gas), thermal decomposition or chemical degradation, leaching of a dispersed phase, volatilization of low boiling materials, by expansion of gas filled materials such as beads or other gas containers, or by a combination of these methods.

[0013] In the second preferred method for creating a foamed hydrocolloid adhesive a mechanical process can be used to add a physical blowing agent, similar to whipping the solid phase into a froth, thus creating a foamed structure. Many processes are possible including incorporation of air, nitrogen, carbon dioxide, or other gases or low boiling point volatile liquids during the compounding, extrusion or converting processes of manufacturing the adhesive.

[0014] Any of the commercially known chemical blowing agents may be used, but the preferred agents will have activation temperatures below about 160°C, preferably with peak gas generation occurring within the range of about 100°C to about 135°C. The chemical blowing agents must also be non-toxic, skin friendly, and environmentally safe, both before and after decomposition.

[0015] The term chemical blowing agent is used herein to cover the use of single or multiple component chemicals in a mixture or paste. Suitable chemical blowing agents include the carbonates of alkali metals, such as ammonium carbonate, ammonium bicarbonate, sodium carbonate, sodium bicarbonate, and calcium carbonate. Improved gas generation can be obtained by making a mixture of carbonates of alkali metals and various organic acids including, but not limited to, stearic, oleic, phthalic, maleic, citric, tartaric acid, and abietic acids. An excess of organic acids are preferably added to the carbonates of alkali metals, so the final reaction products have an acidic character.

[0016] The amount of chemical blowing agent to be added to hydrocolloid adhesive can range from about 0.01% up to about 90% by weight, with a practical range including about 1% up to about 20%. The amount to be added can be determined by measuring the amount of gas generated from a candidate mixture and calculating the amount of foaming required of the final product, tempered by experience of the amount of gas lost to atmosphere during the foaming process.

[0017] One embodiment of a chemical blowing agent is a mixture of sodium bicarbonate and citric acid. The stoichiometric ratio for these two components should be 3 moles of sodium bicarbonate to 1 mole of citric acid. To provide an excess of acid to base, the molar ratio of sodium bicarbonate to citric acid can range from 3 to 1 up to 1 to 3 or higher, preferably near 2 to 1.

[0018] The adhesive mass containing the chemical blowing agent can be processed into a foam under any of the techniques described above under the first preferred method. Processing includes forming the adhesive mass into sheet, either flat or contoured, by extrusion or by pressing or by injection molding or by thermoforming or by any other typical adhesive processing method. The adhesive sheet will be laminated to suitable films and release carriers and then heated sufficiently to cause the decomposition of the chemical blowing agent, thereby creating a foamed hydrocolloid adhesive.

[0019] Cutting of the adhesive sheet into a wound dressing or an ostomy wafer or any other typical hydrocolloid adhesive product shape can be performed either before or after the foaming of the adhesive mass.

[0020] Alternatively, the adhesive mass can be foamed during the extrusion process, followed by calendaring, lamination, and cutting of the foamed sheet into a product.

## Example

[0021] For one example of how a foamed hydrocolloid adhesive may be created, one adhesive formula listed in Patent 4551490 was blended with a chemical blowing agent, shaped into a product, and foamed by addition of heat.

[0022] The base hydrocolloid adhesive formulation used is substantially as described in Patent 4551490 under Example 4. The base hydrocolloid adhesive formulation was manufactured using the adhesive mixing process described in Patent 4551490 under Example 1. The precise adhesive formulation and process used follows.

[0023] The mineral oil, polyisobutylene, SIS rubber, and Antioxidant are combined in a sigma blade mixer with heating (about 115°C.) and agitating for approximately 1.0 to 2.5 hours. The mixture is cooled to about 100°C., and after another 30 minutes of blending, the sodium carboxymethylcellulose, cross-linked sodium carboxymethylcellulose and Tackifier are added. Mixing is continued at about 100°C for 30 minutes until a homogeneous mass is obtained. The homogeneous mass is removed from the mixer and is allowed to cool. The ethylene propylene rubber is then added to the mixer and masticated at 115°C for 10 minutes. The cooled homogeneous mass is then added into the mixer along with the masticated ethylene propylene rubber and allowed to mix for 30 minutes at 115°C or until the entire hydrocolloid adhesive mixture is homogeneous.

Percentage	Ingredient
8.1%	Polyisobutylene, low molecular weight, medium hard
21.8%	Sodium carboxymethylcellulose
8.0%	Cross-linked sodium Carboxymethylcellulose
17.4%	Mineral Oil
17.5%	Styrene-Isoprene-Styrene copolymer rubber
10.9%	Tackifier, pentaerythritol ester of rosin
1.3%	Antioxidant
15.0%	Ethylene Propylene Rubber

[0024] The fully formulated base hydrocolloid adhesive was then heated and masticated in a sigma blade mixer at 80°C for five minutes, in preparation for the addition of the chemical blowing agent. The chemical blowing agent used is the combination of sodium bicarbonate and citric acid at a molar ratio of 2 to 1, added at the 6% by weight level. The chemical blowing agent was then added to the heated and masticated adhesive mass and mixed for twenty minutes until fully dispersed and distributed in the adhesive mass. The adhesive mass with the incorporated chemical blowing agent was then removed from the sigma blade mixer and allowed to cool.

[0025] The prepared adhesive mass was then characterized using a Haake Rheocord equipped with a gas flow meter suitable for measuring chemical blowing agent gas generation over a range of temperatures. Using a temperature sweep with a rate of rise of 1.4°C per minute, gas generation began at 113°C, had a first peak at 124°C and had a second peak at 160°C.

[0026] The prepared adhesive mass was then formed into a sheet using a press set at 80°C, laminated to the desired carrier substrates, and cut into a product shape, in this case a 4" by 4" dressing. Multiple samples of the dressing were then placed into an oven set at various temperatures, for ten minutes each, which activated the decomposition of the chemical blowing agent, thereby creating a foamed hydrocolloid adhesive product.

[0027] The foamed product density varied depending on the foaming temperature. Dressings foamed at the higher temperatures had lower densities and larger cell sizes.

Sample Treatment	Density, g/cc
Not foamed	1.00
135°C	0.85
150°C	0.58
160°C	0.45

[0028] Measurements, all in millimeters diameter, of the cell size distributions for the dressings foamed at various temperatures were as follows:

Temperature	Smallest cell	Typical cell	Maximum cell	Notes
125°C	0.02	0.5	1.0	Closed cells, mostly isolated

(continued)

Temperature	Smallest cell	Typical cell	Maximum cell	Notes
135°C	0.2	0.6	2.0	Some open cells, connected
150°C	0.2	1.6	4.0	Open cells, well connected

[0029] Fluid uptake measurements were performed on unfoamed samples and on samples foamed at 150°C. The fluid uptake measurements used 0.9% aqueous saline as the fluid and were held in a conditioning unit at 38°C, to simulate physiological conditions. Fluid uptake, in grams per square meter, was measured at three different time intervals.

	Average Fluid uptake at each Time interval, g/m <sup>2</sup>		
Condition	1 hour	2 hour	18.5 hours
Not Foamed	179	247	687
Foamed	497	787	1319
Ratio of Uptakes	2.8 times	3.2 times	1.9 times

[0030] As can be seen from the large increase in the uptake of the foamed adhesive compared to the unfoamed adhesive, the effect of foaming the hydrocolloid adhesive will provide improvements in transferring and absorbing moisture, perspiration, and wound fluid. The application of this technology to hydrocolloid adhesives will provide improvement of the performance of the total wound dressing or ostomy appliance.

[0031] For some wounds, it may be desirable to have a dressing that includes a foamed adhesive laminated with a moisture retaining material. This moisture retaining material may include a fibrous or non-fibrous, woven or non-woven, or foamed or non-foamed stock material, or another material possessing capillaries, cells, pores or other channels or compartments for the purpose of retaining or absorbing moisture. This lamination material may be combined with a moisture retention aid including hydrocolloids, super absorbents, or other liquid gelling, liquid absorbing, or liquid retaining components for retaining or controlling liquid and moisture.

[0032] It may also be desirable to laminate the foamed adhesive with an occlusive layer for the purpose of retaining moisture or managing transmission.

[0033] It will be appreciated that the foregoing description is merely illustrative of preferred forms of the invention, and that many modifications may be included within the scope of the invention.

### Claims

1. A process for manufacturing an absorbent adhesive, the process comprising: forming a mass of pressure-sensitive hydrocolloid adhesive; introducing gas generating material into the mass of hydrocolloid adhesive; and generating gas bubbles in the adhesive by activating the gas generating material.
2. A process according to claim 1, further comprising the step of shaping the mass of hydrocolloid adhesive into a predetermined shape.
3. A process according to claim 2, wherein the step of shaping the mass is performed prior to the step of generating the gas bubbles.
4. A process according to claim 1, 2 or 3, wherein the gas generating material includes a chemical blowing agent.
5. A process according to claim 4, wherein the chemical blowing agent includes a carbonate.
6. A process according to claim 5 wherein the carbonate is one or more compounds selected from ammonium carbonate, ammonium bicarbonate, sodium carbonate, sodium bicarbonate and calcium carbonate.
7. A process according to claim 1, 2 or 3, wherein the gas generating material includes an acid.
8. A process according to claim 7, wherein the acid is one or more compounds selected from stearic, oleic, phthalic, maleic, citric, tartaric and abietic acids.

9. A process according to claim 4, wherein the chemical blowing agent includes a mixture of sodium bicarbonate and citric acid.
- 5 10. A process according to claim 4 or any claim dependent thereon, wherein the chemical blowing agent has a range of approximately 1% to 20% by weight.
11. A process according to any preceding claim, wherein the step of shaping includes making said adhesive into a sheet.
- 10 12. A process according to any preceding claim, further comprising the step of laminating at least one layer onto said predetermined shape.
13. A process according to claim 12, further comprising the step of laminating a non'. occlusive or occlusive layer onto said predetermined shape.
- 15 14. A process according to any preceding claim, wherein the step of generating gas bubbles includes incorporating a chemical blowing agent into the hydrocolloid adhesive and heating the blowing agent.
- 20 15. A process according to any proceeding claim, wherein the gas bubbles include one or more of air, nitrogen and carbon dioxide.
- 25 16. A process for manufacturing an absorbent adhesive, the steps comprising: forming a mass of a pressure-sensitive hydrocolloid adhesive; introducing gas bubbles into the mass of hydrocolloid adhesive; and forming the adhesive into a predetermined shape.
17. A process according to claim 16, where the step of introducing includes mechanically introducing.
18. A process according to claim 17, where the step of mechanically introducing includes whipping the mass into a froth.
- 30 19. Absorbent adhesive for attachment to the body, comprising a pressure-sensitive hydrocolloid adhesive, said hydrocolloid adhesive including gas bubbles or cells incorporated therein, said hydrocolloid adhesive being shaped for application to the skin.
- 35 20. An adhesive according to claim 19, wherein said gas bubbles include one or more of air, nitrogen and carbon dioxide.
21. An adhesive according to claim 19 or 20, wherein said gas bubbles are cells distributed through said hydrocolloid adhesive having a diameter in the range of .2 to 4.0 millimeters.
- 40 22. An adhesive according to claim 19, 20 or 21, wherein said hydrocolloid adhesive with gas bubbles incorporated is 45 to 85% of the density of the hydrocolloid adhesive without gas bubbles predeterminedly incorporated therein.
- 45 23. An adhesive according to any of claims 19 to 22, wherein said hydrocolloid adhesive with gas bubbles predeterminedly incorporated therein uptakes .9% aqueous saline at 38°C in grams per square meter in one hour at more than 2 times the hydrocolloid adhesive without gas bubbles predeterminedly incorporated therein.
24. Absorbent adhesive for attachment to the body, the adhesive comprising a foamed hydrocolloid adhesive having gas bubbles or cells incorporated within the hydrocolloid adhesive mass.
- 50 25. A wound dressing comprising an adhesive as defined in any of claims 19 to 24.
26. An ostomy device comprising an adhesive as defined in any of claims 19 to 24.
- 55 27. An attachment device for attaching an ostomy appliance to a wearer's body, the device comprising an adhesive as defined in any of claims 19 to 24.

European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number  
EP 00 30 1693

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.7)
X	US 5 089 606 A (COLE SUSAN M ET AL) 18 February 1992 (1992-02-18) * claims; examples * ---	1,4-10	A61L15/00 A61L15/28 A61L15/58 C08J9/30
X	WO 94 00512 A (ALBANY INT CORP ;BAKIS GEORGE (US); EAGLES DANA BURTON (US); HAG00) 6 January 1994 (1994-01-06) * page 7; claims; examples * ---	16-22, 24-27	
Y	DE 44 18 818 A (STOCKHAUSEN CHEM FAB GMBH) 12 January 1995 (1995-01-12)  * page 4, line 36 - line 43; claims; examples * ---	1,4-7, 10, 14-16, 19-27	
Y	DE 196 20 109 A (BEIERSDORF AG) 20 November 1997 (1997-11-20)  * page 2, line 43 - line 56 * ---	1,4-7, 10, 14-16, 19-27	TECHNICAL FIELDS SEARCHED (Int.Cl.7)
Y	US 4 551 490 A (DOYLE ARTHUR ET AL) 5 November 1985 (1985-11-05)  * column 2, line 27 - line 64 * * column 4, line 8 - line 28; claims; examples * ---	1,4-7, 10, 14-16, 19-27	A61L C08J
A	EP 0 818 187 A (HOLLISTER INC) 14 January 1998 (1998-01-14) * claims; figures * -----	2,3, 11-13	
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 8 May 2000	Examiner Baumschabl
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons @ : member of the same patent family, corresponding document	

EPO FORM 1503 (03/02) (P0/C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.**

EP 00 30 1693

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.  
The members are as contained in the European Patent Office EDP file on  
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

08-05-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5089606 A	18-02-1992	US 5057606 A	15-10-1991
		US 4948575 A	14-08-1990
		CA 2006882 A	24-07-1990
		DE 69010221 D	04-08-1994
		DE 69010221 T	26-01-1995
		EP 0380254 A	01-08-1990
		JP 2232234 A	14-09-1990
		CA 2008441 A	24-07-1990
		DE 69007134 D	14-04-1994
		DE 69007134 T	06-10-1994
		EP 0380253 A	01-08-1990
		JP 2234758 A	17-09-1990
WO 9400512 A	06-01-1994	AU 672214 B	26-09-1996
		AU 4543793 A	24-01-1994
		BR 9305552 A	20-12-1994
		CA 2116037 A	06-01-1994
		EP 0612331 A	31-08-1994
		FI 940735 A	16-02-1994
		JP 2644626 B	25-08-1997
		JP 6510330 T	17-11-1994
		NO 940546 A	17-02-1994
		US 5840777 A	24-11-1998
		US 5851461 A	22-12-1998
DE 4418818 A	12-01-1995	AT 157674 T	15-09-1997
		AU 3320799 A	26-08-1999
		AU 683291 B	06-11-1997
		AU 7383894 A	06-02-1995
		BR 9406938 A	10-09-1996
		CA 2166779 A	19-01-1995
		DE 59403981 D	09-10-1997
		EG 20478 A	31-05-1999
		WO 9502002 A	19-01-1995
		EP 0707603 A	24-04-1996
		ES 2087840 T	01-08-1996
		FI 960083 A	08-01-1996
		GR 96300041 T	31-08-1996
		HU 74317 A,B	30-12-1996
		IL 110134 A	15-07-1998
		JP 2849617 B	20-01-1999
		JP 8509521 T	08-10-1996
		US 5712316 A	27-01-1998
DE 19620109 A	20-11-1997	AU 719280 B	04-05-2000
		AU 2773097 A	09-12-1997

EPO FORM P0119

For more details about this annex ; see Official Journal of the European Patent Office, No. 12/82



ANNEX TO THE EUROPEAN SEARCH REPORT  
ON EUROPEAN PATENT APPLICATION NO.

EP 00 30 1693

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

08-05-2000

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 19620109 A		WO 9743992 A	27-11-1997
		EP 0901356 A	17-03-1999
US 4551490 A	05-11-1985	AT 36808 T	15-09-1988
		AU 578612 B	03-11-1988
		AU 2950184 A	10-01-1985
		CA 1225764 A	18-08-1987
		DD 223459 A	12-06-1985
		DE 3473731 D	06-10-1988
		DK 311384 A,B,	28-12-1984
		EP 0130061 A	02-01-1985
		ES 533717 D	01-12-1985
		ES 8602898 A	16-03-1986
		IE 58125 B	14-07-1993
		IL 72163 A	30-10-1987
		JP 1970283 C	18-09-1995
		JP 6078507 B	05-10-1994
		JP 60020976 A	02-02-1985
		NO 842584 A,B,	28-12-1984
		NZ 208566 A	12-11-1986
		ZA 8404601 A	27-02-1985
EP 0818187 A	14-01-1998	US 5935363 A	10-08-1999

EPO FORM 10453

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82